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Effect of Hot-Air and Freeze-Drying on the Quality Attributes of Dried Pomegranate (*Punica granatum* L.) Arils During Long-Term Cold Storage of Whole Fruit

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Abstract: This study investigated the effect of hot-air and freeze-drying on the physicochemical, phytochemical and antioxidant capacity of dried pomegranate arils during long-term cold storage (7 ± 0.3 °C, with 92 ± 3% relative humidity) of whole fruit over a single experiment. Extracted arils were processed at monthly intervals during 12 weeks of cold storage of whole fruit. After the 12-week storage period, hot-air and freeze-dried arils showed the least (3.02) and highest (23.6) total colour difference (TCD), respectively. Hot-air dried arils also contained 46% more total soluble solids (TSS) than freeze-dried arils. During the storage of pomegranate fruit, total phenolic content (TPC) steadily increased from 20.9 to 23.9 mg GAE/100 mL and total anthocyanin content (TAC) increased from 6.91 to 8.77 mg C3gE /100 mL. Similarly, an increase in TPC and TAC were observed for hot-air (9.3%; 13%) and freeze-dried arils (5%; 5%), respectively. However, the radical scavenging activity (RSA) reduced by 8.5 and 17.4% for hot-air and freeze-dried arils, respectively, after 12 weeks of cold storage. Overall, the parameters such as colour, TPC and TAC as well as the lower degradation in RSA stability during storage showed distinct differences in quality when using the freeze-drying method, which is, therefore, recommended.

Keywords: cold storage; fresh arils; dried methods; total soluble solids; total phenolic content; storage stability

1. Introduction

Pomegranate fruit (*Punica granatum* L.) is renowned for its bioactive phenolic content, including flavonoids, phenolic acids, tannins, ellagitannins, catechin, rutin and epicatechin [1,2]. These antioxidants have been implicated in the protection against heart, cancer, immune system and other chronic diseases [3,4]. South Africa is leading the pomegranate production and export in the Southern Hemisphere, with an estimated production of 540,000 tonnes/1,200,000 cartons [5]. However, 11% of the total production is processed locally, and 9% is considered as waste due to disorders such as cracks, sunburn, scalds and bruises, which could affect the internal quality of the fruit [5,6]. Fruit similar to pomegranate usually has a small harvest window, whereas processing is carried out over a long period, and this requires the storage of raw materials for the production of niche products. Fifty per cent of fruit that do not meet export requirements are often converted into products such as jellies and juices, which have a short shelf life [7]. Caleb et al. [8] reported a maximum flavour-life of seven days for

pomegranate arils. However, drying is a preservation method that reduces the moisture contained in food, thereby extending the shelf-life of the product [9].

Dried pomegranate arils are often referred to as 'anardana' and are used in many traditional medicinal formulations to treat neurological and kidney disorders, as well as stomach and cardiac infections [10]. Due to its acidity profile, these dried arils help to improve digestion and mouth-feel [11]. Indian and Pakistani cuisines use 'anardana' as a condiment, but it can also be used as a substitute for tamarind and mango powder, or in culinary preparations of fruit salad, flavoured yoghurt and ice cream [12]. However, different drying methods, packaging and storage conditions are major factors affecting the inherent characteristics of the final product [13].

In addition to the decline in quality of pomegranate fruit during storage, different processing techniques could also have a negative impact on the quality of the finished product. Previous studies suggest that freeze-drying retains more bioactive compounds during the processing of fruit in comparison to other drying methods. For instance, Asami et al. [14] reported higher retention of phenolic concentration in 'Marion' blackberries during freeze-drying than hot-air drying. Shofian et al. [15] reported that the low temperature used to withdraw water from fruit material in freeze-drying helped to preserve the antioxidant capacity of tropical fruits. However, the freeze-drying process could be expensive and energy-consuming [16]. Among several drying methods available, hot-air drying is cost- and energy-efficient, making it one of the most commonly used methods for drying food materials [17]. However, it has a greater effect on the deformation of final products which is often characterised by dislocation of volatile substances and changes in physical properties [18].

'Wonderful' pomegranate is the most cultivated and consumed globally [19,20]. Currently, there has been a considerable rise in the export of pomegranate fruit grown in South Africa, with an estimated at approximately 70% of total production [21] compared to 56% export in 2013 [22]. 'Wonderful' is desirable because its bioactive compounds are better maintained during a prolonged storage duration compared to other cultivars. According to Arendse [23], the quality attributes of 'Wonderful' were maintained over five months in storage. Furthermore, the highest scavenging capacity exhibited by 'Wonderful' compared to the other eight pomegranate cultivars suggests the commercial potential of the cultivar [24].

The concentration of bioactive compounds in dried fruit products is influenced by numerous factors, including cultivar, harvest maturity, processing method and storage conditions [25]. While there are several studies on the effect of cultivar [26,27], there is a dearth of information on the impact of harvest maturity, extended storage of raw material and processing method [28] on the bioactive compounds of dried fruit, including dried pomegranate aril. In practice, fruit are kept in storage to allow processing at intervals based on demand or processing capacity. Fruit quality attributes degrade over time and will affect the quality of processed products, and hence, important to establish the maximum holding time of raw materials before processing. Therefore, this study aimed to examine the effects of hot-air and freeze-drying on the quality attributes of dried pomegranate arils during prolonged cold storage of whole fruit (raw material).

2. Materials and Methods

2.1. Fruit Supply and Storage Condition

Pomegranate fruit (cv. Wonderful) was handpicked at commercial harvest period from Blydeverwacht orchard in Wellington, (latitude 33°01′00″ S, longitude 18°58′59″ E) Western Cape Province, South Africa during the 2018/2019 growing season. Fruit were transported in an air-conditioned vehicle to the Postharvest Technology Research Laboratory at Stellenbosch University. Fruit without visible external discolouration or injuries were sorted to include fruit of uniform colour and size. After sorting, fresh fruit were packed inside standard open top cartons with the following dimensions: width 0.3 m, length 0.4 m, height 0.133 m and a total of 22 perforations and stored at 7 ± 0.3 °C, with 92 ± 3% relative humidity (RH). Fruit were sampled at 0, 4, 8 and 12 weeks as described in the experimental flow chart (Figure 1).

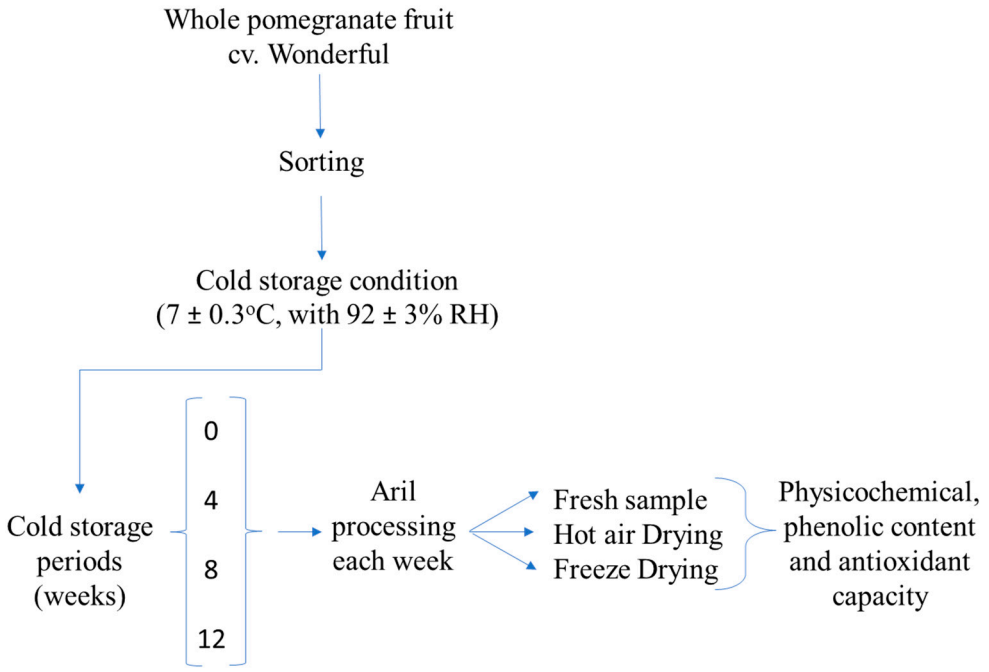

Figure 1. Shows a description of the experimental flowchart.

Temperature (°C) and relative humidity (% RH) within the cold rooms were taken every hour throughout storage. This was carried out with the use of a Tiny Tag TV-4500 data loggers (Gemini Data Logger, Sussex, UK) with a functional range of −40 °C to +85 °C and 0% to 100% RH.

2.2. Characterisation of Fresh Arils

Fresh pomegranate arils were periodically evaluated before processing for total soluble solids (TSS) by a refractometric method and titratable acidity (TA) by titrating to pH 8.1 with 0.1 N NaOH. Additionally, moisture content was measured using a digital moisture analyser. The Folin-Ciocalteu method was used to quantify the total phenolic content (TPC) and expressed as mean ± SE (milligram) gallic acid equivalent (GAE) per 100 mL of crude juice, while the pH differential method was used to determine the total anthocyanin content (TAC) [1,29], which was expressed as mean ± SE (milligrams) cyanidin-3-glucoside per 100 mL of crude juice. The antioxidant capacity (radical scavenging activity, RSA; ferric ion reducing antioxidant power, FRAP) was also measured in triplicate, according to Fawole and Opara [30] and expressed as Trolox equivalent (mM) per 100 mL of crude juice.

2.3. Drying Procedure

2.3.1. Freeze-Drying

Arils were placed in a freeze-drying paper bag and frozen in a static air freezer at −80 °C. Frozen samples were freeze dried in triplicates. The specimen jar containing samples were carefully taken to a laboratory-scale freeze-dryer (VirTis Co., Gardiner, NY, USA) operating at condenser temperature −85 °C and pressure 45 mTorr. Sample weight was measured every third hour until no change in weight was detected, which was after 96 h.

2.3.2. Hot-Air Drying

Arils were dried at 60 °C in a hot-air oven for 11 h to achieve a 10–12% moisture content. Constant air velocity was maintained at 1.0 m s^{-1} for each treatment. To ensure an inner temperature of 60 °C was reached, the hot-air dryer was switched on at least an hour before drying, and the temperature was confirmed using a thermometer, before spreading the arils in glassy Petri dishes and placing them in the drying chamber. Dried arils were packed and sealed in food-grade moisture-resistant plastic

bags and stored in glass desiccators containing calcium sulphate (Sigma-Aldrich Pty. Johannesburg, South Africa).

2.4. Colour Measurement

By the direct reading using a chromo-meter (Minolta model CR-200, Osaka, Japan), dried aril colour was determined to obtain the colour values: L^* (brightness/darkness), a^* (redness/greenness), C* (colour intensity) and h° (colour purity). The measurements were recorded at three different times from a transparent petri dish and averaged. The maximum for L^* value is 100 (white), and the minimum is zero (black). Positive a^* value is red and negative a^* is green, while positive b^* value is yellow and negative b^* is blue. The total colour difference (TCD) was calculated [4,31] as:

$$TCD = \left[(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\right]^{\frac{1}{2}} \quad (1)$$

where ΔL^*, Δa^* and Δb^* represents the value before and after drying at each treatment levels and results were expressed as means ± S.E.

2.5. Characterisation of Dried Arils

Dried pomegranate arils were ground into powder using liquid nitrogen followed by extraction of 5 g sample in 50 mL of distilled water. For 5 min the mixture was vortexed and sonicated for 15 min in an ultrasonic bath. This was followed by centrifugation at 10,000 rpm for 25 min and recovery of the supernatant for TSS, TA and pH measurements. For phytochemical properties and antioxidant capacity, the same extraction procedure was followed using 50% methanol.

2.6. Chemical Properties

Total Soluble Solids and Titratable Acidity Determination

TSS was estimated using a digital hand refractometer (model PT-32; ATAGO, Tokyo, Japan) with the range of 0–32 °Brix, which was blanked with distilled water. For TA, 2 mL of the supernatant was diluted in seventy millilitres of distilled water and titrated against 0.2 N of sodium hydroxide (NaOH) to a pH of 8.2 using a Metrohm 862 Compact titrosampler (Herisau, Switzerland).

2.7. Determination of Phytochemical Properties

2.7.1. Total Phenolic Content (TPC)

Folin–Ciocalteu method using a methanolic extract of dried arils was used to determine the TPC [30]. A 0.05 mL of the supernatant was mixed with 0.45 mL of 50% methanol in a test tube followed by adding 0.5 mL Folin–Ciocalteu after 2 min. The mixture was then vortexed and kept in the dark for 10 min before adding 2% Na_2CO_3 and further incubated for 40 min in the dark. The absorbance of each sample was read at 520 nm in a UV-visible spectrophotometer (Thermo Scientific technologies, Madison, USA) against a blank containing 50% methanol. Absorbance was compared with a standard curve (Gallic acid, 0–10 mg), and results were expressed as mg gallic acid equivalent per gram pomegranate dry matter (mg GAE/g DM).

2.7.2. Total Anthocyanin Content

By using the pH differential method, total anthocyanin content (TAC) was quantified [29]. In triplicates, 1 mL of extract was separately mixed with 9 mL of pH 1.0 and pH 4.5 buffers. Absorbance was measured at 520 and 700 nm in pH 1.0 and 4.5 buffers, and the result was expressed as cyanidin 3-glucoside using Equations (2) and (3):

$$A = (A_{510} - A_{700})_{\text{pH 1.0}} - (A_{510} - A_{700})_{\text{pH4}} \quad (2)$$

$$\text{Total monomeric anthocyanin}(\text{mg/mL}) = \frac{\text{A} \times \text{MW} \times \text{DF}}{\varepsilon \times \text{L}} \quad (3)$$

where A = Absorbance, ε = Cyd-3-glucoside molar absorbance (26,900), MW = anthocyanin molecular weight (449.2), DF = dilution factor and L = cell path length (1 cm). Results are expressed as equivalent per gram dry matter (mg C_3gE/g DM).

2.8. Antioxidant Capacity

2.8.1. Radical-Scavenging Activity (RSA)

The RSA was quantified in triplicate, according to Fawole et al. [30]. Aqueous methanolic extract of dried aril (0.015 mL) was diluted with methanol (0.735 mL) in test tubes, briefly under dim light shade, followed by adding 0.75 mL, 0.1 mM methanolic DPPH solution. For 30 min in the dark and at room temperature, the mixtures were incubated, and the absorbance was measured at 517 nm using a UV-vis spectrophotometer (Thermo Scientific technologies, Madison, USA). Absorbance was compared with the standard curve (Trolox equivalent, 0–2.0 mM). The free-radical activity of dried aril was expressed as Trolox equivalent (mM) equivalent per gram dry matter (mM TE/g DM).

2.8.2. Ferric Ion Reducing Antioxidant Power (FRAP)

The antioxidant power of dried aril was measured using the colourimetric method according to [30,32]. The FRAP working solution was freshly prepared in mixtures of 300 mM acetate buffer (50 mL), 10 mM 2,4,6-tripyridyl-s-triazine (TPTZ) (5 mL) and 20 mM ferric chloride (5 mL) at 37 °C. In triplicates, diluted aqueous methanolic dried aril extracts (0.15 mL) were added to 2.85 mL of the FRAP working solution followed by a 30 min incubation in the dark. By measuring the absorbance at 593 nm, the reduction of the Fe^{3+}-TPTZ complex to a coloured Fe^{2+}-TPTZ complex at low pH of dried aril extracts was monitored. Trolox (0–10 mM) was used for the calibration curve, and the results were expressed as Trolox (mM) equivalents per gram dry matter (mM TE/g DM).

2.8.3. Stability of RSA and FRAP

Several studies have reported the use of simple first-order reaction kinetics to describe storage and thermal degradation of bioactive compounds from various sources. Li et al. [33] and Moldovan et al. [34] described the degradation kinetics as in Equation (4):

$$\ln[\text{RSA}] = \ln[\text{RSA}_0] - k\text{t} \quad (4)$$

where RSA = antioxidant capacity, mM TE/g dried aril at time t; RSA_0 = initial RSA, mM TE/g; k = reaction rate constant, weeks^{-1}; t = reaction time, weeks. The half-life of antioxidant capacity from the investigated extracts during storage can be calculated using Equation (4):

$$\text{t}_{1/2} = -\ln 0.5/k \quad (5)$$

where $\text{t}_{1/2}$ = half-life (weeks) and k = reaction rate constant (weeks^{-1}).

2.9. Statistical Analysis

The measurement made from chemical properties, colour and phytochemical properties were subjected to statistical evaluation. STATISTICA (Statistica 13.0, StatSoft Inc., Tulsa, OK, USA) was used to process the data and expressed as means ± standard error. All analysis was done in triplicates. For fresh aril characterisation, data were subjected one-way analysis of variance (ANOVA) and for dried aril characterisation with different drying methods, data were subjected to two-way ANOVA. Means were separated according to Fisher's LSD test at a level of significance of 95%. The graphs were presented using GraphPad Prism software 4.03 (GraphPad Software, Inc., San Diego, CA,

USA), while the XLSTAT software version 1 April 2012 (Addinsoft, France) was used to estimate Pearson's correlation.

3. Results

3.1. Effect of Cold Storage on Moisture Content of Pomegranate Aril

The moisture content of fresh pomegranate arils decreased gradually with storage time from 74.7% to 57.4% (Table 1), which affected the weight of the fruit. Pomegranate fruit has been reported to be highly susceptible to weight loss [35], which lead to the visible dehydration observed in Figure 2. The reduced weight observed during storage could be attributed to transpiration through large pores in the fruit peel [4,36]. The reduction in the weight of the whole fruit consequently resulted in a weight reduction of the arils. These findings were corroborated by Fawole and Opara [4], who reported a significant reduction in weight of pomegranate fruit during cold storage.

Table 1. Changes in physicochemical attributes of fresh pomegranate arils during 12 weeks of cold storage at 7 ± 0.3 °C, 92 ± 3% RH (wet basis, w.b).

Storage Period (Weeks)	Moisture Content (%)	TSS (°Brix)	TA (% Citric Acid)	TSS:TA	TCD
0	74.7 ± 1.25 ^{a}	13.7 ± 0.25 ^{c}	0.38 ± 0.03 ^{a}	36.7 ± 2.01 ^{c}	-
4	71.9 ± 0.92 ^{a}	14.4 ± 0.22 ^{b}	0.33 ± 0.01 ^{ab}	44.2 ± 2.25 ^{c}	5.69 ± 1.18 ^{b}
8	67.8 ± 0.73 ^{b}	14.8 ± 0.05 ^{ab}	0.28 ± 0.01 ^{bc}	53.2 ± 2.10 ^{b}	4.31 ± 0.77 ^{b}
12	57.4 ± 1.08 ^{c}	15.1 ± 0.06 ^{a}	0.24 ± 0.01 ^{c}	62.5 ± 2.97 ^{a}	11.2 ± 1.43 ^{a}

TSS, total soluble solids; TA, titratable acidity; TCD, total colour difference. Data presented as means ± SE in each column followed by different letters are significantly different ($p < 0.05$) according to Fisher's LSD.

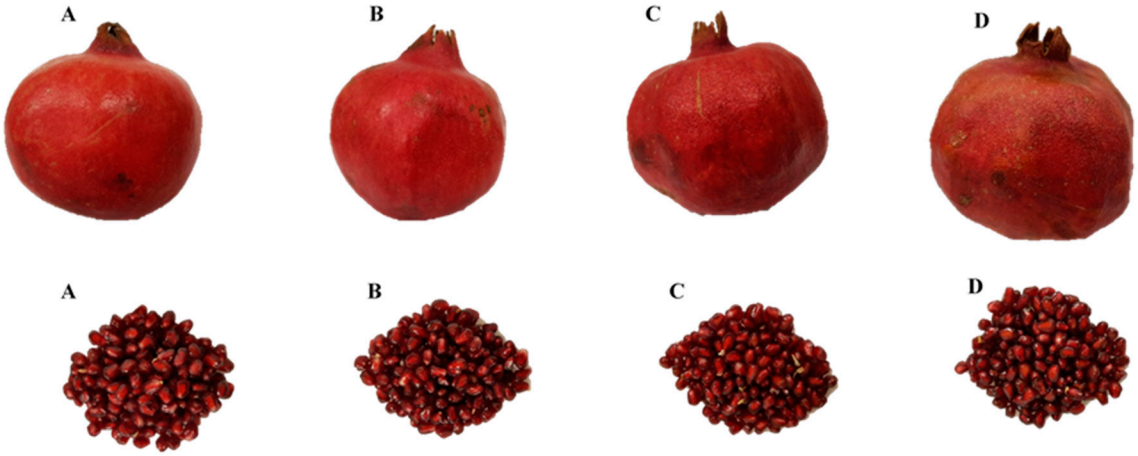

Figure 2. Pomegranate whole fruit (raw material) at harvest **(A)** and during cold storage at 7 ± 0.3 °C, 92 ± 3% RH (w.b) at 4 weeks (**B**), 8 weeks (**C**) and 12 weeks (**D**) storage period. Fresh pomegranate arils show no noticeable differences visible to the naked eye for the period of 12 weeks.

Visual browning of 5% was observed in the arils immediately after peeling the fruit after eight weeks of storage, gradually increasing to 15% at the 12-week storage period. However, differences in the arils over time were unnoticeable in the pictorial representation in Figure 2. A similar study by Konopacka and Plocharski [37] reported increasing tissue browning in apple subjected to long term storage. Conversely, chemical dipping of 'Taify' pomegranate fruit before cold storage showed no browning of the aril tissue [38].

3.2. TCD of Fresh and Dried Pomegranate Arils

Storage of pomegranate fruit contributed to the changes in the TCD of fresh arils, and subsequently had a significant effect on the TCD of dried arils. A notable variation was observed in the TCD with increased storage period, with the highest TCD being 11.2 after the 12-week storage period (Table 1). For dried arils processed with hot-air and freeze-dryers, there was a significant ($p < 0.0001$) interaction in TCD (Table 2). Hot-air drying had the least (3.02), while freeze-dried arils had the highest (23.6) TCD after the 12-week storage period (Table 2). A change in TCD is an important attribute of a dried product, expressing the capacity of the human eye to distinguish between various colours attributed to different products [27]. Coklar et al. [39] reported similar findings where hawthorn fruit dried using a freeze dryer had a better colour appearance than fruit dried with oven and microwave dryers. Ali et al. [40] reported that freeze-dried guava fruit preserved its colour the best compared to sunlight and convective oven dryer. The colour change of dried arils could be influenced by the drying method involved and also by the naturally occurring biochemical changes happening during storage of pomegranate fruit.

Table 2. Changes in the physicochemical properties of dried pomegranate arils during 12 weeks of cold storage at 7 ± 0.3 °C, 92 ± 3% RH (w.b).

Drying Method	Storage Period (Weeks)	TCD	TSS (°Brix)	TA (% Citric Acid)	TSS:TA
Hot-air drying	0	-	22.2 ± 0.67 ^{a}	3.15 ± 0.17 ^{b}	7.03 ± 0.19 ^{c}
	4	7.15 ± 0.86 ^{b}	22.7 ± 0.73 ^{a}	3.23 ± 0.01 ^{a}	7.00 ± 0.21 ^{c}
	8	1.81 ± 0.71 ^{c}	23.7 ± 0.44 ^{a}	3.13 ± 0.00 ^{bc}	7.55 ± 0.15 ^{c}
	12	3.02 ± 1.09 ^{bc}	23.5 ± 0.58 ^{a}	3.10 ± 0.02 ^{c}	7.58 ± 0.22 ^{c}
Freeze-drying	0	-	17.5 ± 1.00 ^{b}	1.14 ± 0.01 ^{e}	15.4 ± 0.86 ^{a}
	4	19.6 ± 2.77 ^{a}	15.0 ± 0.29 ^{c}	1.20 ± 0.01 ^{d}	12.5 ± 0.36 ^{b}
	8	3.94 ± 1.32 ^{bc}	14.0 ± 0.50 ^{cd}	1.24 ± 0.03 ^{d}	11.3 ± 0.62 ^{b}
	12	23.6 ± 2.55 ^{a}	12.8 ± 0.33 ^{d}	1.14 ± 0.01 ^{e}	10.2 ± 0.36 ^{b}
Drying method (A)		0.0001	0.0001	0.0001	0.0001
Storage period (B)		0.0001	0.0910	0.0001	0.0020
A × B		0.0001	0.0007	0.0060	0.0002

TSS, total soluble solids; TA, titratable acidity; TCD, total colour difference. Data presented as means ± SE in each row followed by different letters are significantly different ($p < 0.05$) according to Fisher's LSD.

3.3. Total Soluble Solids (TSS) and Titratable Acidity (TA) of Fresh and Dried Arils

The investigated chemical attributes (TSS and TA) in fresh pomegranate arils were significantly ($p < 0.05$) different from those measured after a period of storage (Table 1). For instance, the TSS of fresh aril increased from 13.7 to 15.1 °Brix after storage (Table 1), while the TA decreased from 0.38 to 0.24 at 12 weeks' storage. In agreement with our study, Arendse et al. [6] reported that pomegranate cultivar Wonderful stored at 5 °C showed an increase in TSS as the storage period progressed. A decrease in TA could be attributed to organic acid break down during the storage period [41]. Fawole and Opara [30] also observed a decrease in TA values for two South African grown cultivars, Bhagwa and Ruby, due to the ongoing metabolism in the fruit during storage.

In dried arils, all chemical attributes showed significant ($p < 0.0001$) interactions with storage period and drying methods (Table 2). Total soluble solids gradually increased with storage period in the hot-air dried arils to almost twice the amount of TSS in freeze-dried arils after storage. The high TSS value could be attributed to drying under high temperature, which resulted in the caramelisation of the product [42].

Throughout the trial, TA was more than double in arils processed with hot-air (3.10–3.15% citric acid) compared to freeze-dried arils (1.14–1.24% citric acid); this could be attributed to the different drying temperatures used (Table 2). Titratable acidity increased after four and eight weeks in hot-air and freeze-dried arils, respectively, before declining with prolonged storage. Ashebir et al. [43] also

noted a significant change in the TSS and TA concentrations of dried tomatoes due to variations in the level of drying temperatures.

The TSS:TA ratio is a good indication of flavour and used as one of the quality indexes of pomegranate fruit [44]. Opposite trends of TSS:TA were observed in dried arils after storage—a slight increase from 7.0 to 7.58 in hot-air dried arils and a significant decrease from 15.4 to 10.2 in freeze-dried arils (Table 2). This implies that storage followed by higher temperature drying enhances the caramelisation and Maillard reaction, breaking down the disaccharides into monosaccharides, and seemingly increasing the TSS content in pomegranate. TSS:TA values ranged between in hot-air dried arils and freeze-dried arils. Higher TSS:TA values observed in freeze-dried arils compared to hot-air dried arils reflect a higher percentage of sugar to acid ratio in dried aril.

3.4. Total Phenolic Content (TPC) and Total Anthocyanin Content (TAC) of Fresh and Dried Arils

During storage of pomegranate fruit, a steady increase in both TPC (from 20.9 to 23.9 mg GAE/100 mL) and TAC (from 6.91 to 8.77 mg C3gE /100 mL) was observed (Table 3). Arendse et al. [6] reported a similar increase in TPC of pomegranate arils cv. 'Wonderful' stored at 5 °C, 7.5 °C and 10 °C for 5 months. Labbe et al. [45] also reported an increase in the total phenolic content of 'Chilean Chaca' pomegranate cultivar at 5 °C for 12 weeks.

Table 3. Changes in the phytochemical properties and antioxidant capacity of fresh pomegranate arils during 12 weeks of cold storage at 7 ± 0.3 °C, 92 ± 3% RH (w.b.).

Storage Period (Weeks)	TPC mg GAE/100 mL	TAC Cyanidin-3-Glucoside (mg/100 mL)	RSA mM TE/100 mL	FRAP mM TE/100 mL
0	20.9 ± 6.27 ^{c}	6.91 ± 3.11 ^{c}	12.4 ± 1.66 ^{a}	2.36 ± 0.36 ^{a}
4	22.1 ± 0.59 ^{b}	7.56 ± 4.88 ^{bc}	10.4 ± 1.66 ^{b}	2.27 ± 0.05 ^{a}
8	22.9 ± 0.65 ^{ab}	8.44 ± 1.62 ^{ab}	8.40 ± 1.71 ^{c}	2.09 ± 0.34 ^{b}
12	23.9 ± 2.35 ^{a}	8.77 ± 0.37 ^{a}	4.92 ± 1.79 ^{d}	2.07 ± 0.68 ^{b}

RSA, radical scavenging activity; FRAP, ferric reducing antioxidant power; TPC, total phenolic content; TAC, total anthocyanin content; w.b. wet basis; Data presented as means ± SE in each column followed by different letters are significantly different ($p < 0.05$) according to Fisher's LSD.

Anthocyanin compounds exhibit the main characteristic red colour in pomegranate fruit [35]. Increase in anthocyanin concentration during storage could be related to the increase in biosynthesis and accumulation of anthocyanin, which is induced at lower temperatures in pomegranate fruit [46]. Results from this study agree with those reported by Arendse et al. [6], who attributed an increase in TAC in pomegranate 'Wonderful' to the continued accumulation of anthocyanins at lower temperatures during storage.

After 12 weeks of cold storage, TPC increased, albeit insignificantly, from 105.9 to 116.7 mg GAE/g DM in hot-air dried pomegranate arils, and from 135.6 to 142.7 mg GAE/g DM in freeze-dried arils. Drying methods contributed to the retention of TPC ($p < 0.0001$), as shown in Figure 3. The freeze-drying method retained approximately 18.2% more TPC than hot-air dried arils. This is in support of the study by Shishehgarha et al. [47], who reported that the freeze-drying method is a precision technology utilised to produce high-quality dried products. Additionally, the increased TPC in freeze-dried pomegranate arils could be attributed to mild fruit cell destruction during freezing and ice sublimation, which consequently enhances extraction of biochemical components [14].

The combined effect of drying method ($p < 0.0001$) and storage period ($p < 0.003$) influenced retention of TAC (Figure 4). This figure shows an increase throughout the 12-week storage period of approximately 13 and 5% in TAC of hot-air and freeze-dried pomegranate arils, respectively. A similar trend was observed in fresh arils during cold storage. However, the TAC of freeze-dried arils was higher compared to hot-air dried arils. This is in agreement with other authors who reported higher anthocyanin content in freeze-dried compared to hot-air dried blackberries [13] and blueberries [48].

The vacuum pressure combined with minimal temperature used during the freeze-drying process preserves bioactive compounds from oxidation [13,48].

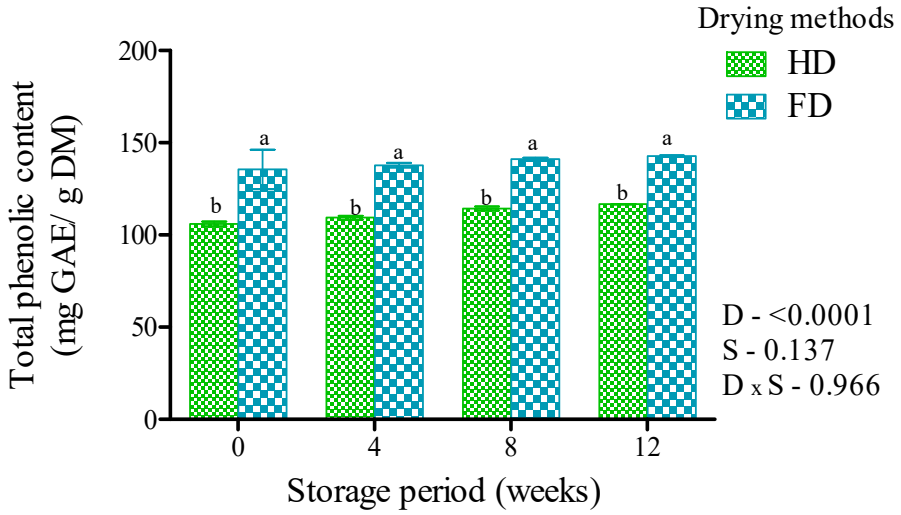

Figure 3. Changes in the total phenolic content of pomegranate dried arils during 12 weeks of cold storage at 7 ± 0.3 °C, 92 ± 3% RH (w.b). HD, hot-air drying; FD, freeze-drying. D, drying methods; S, storage period (week). Different letters are significantly different ($p < 0.05$).

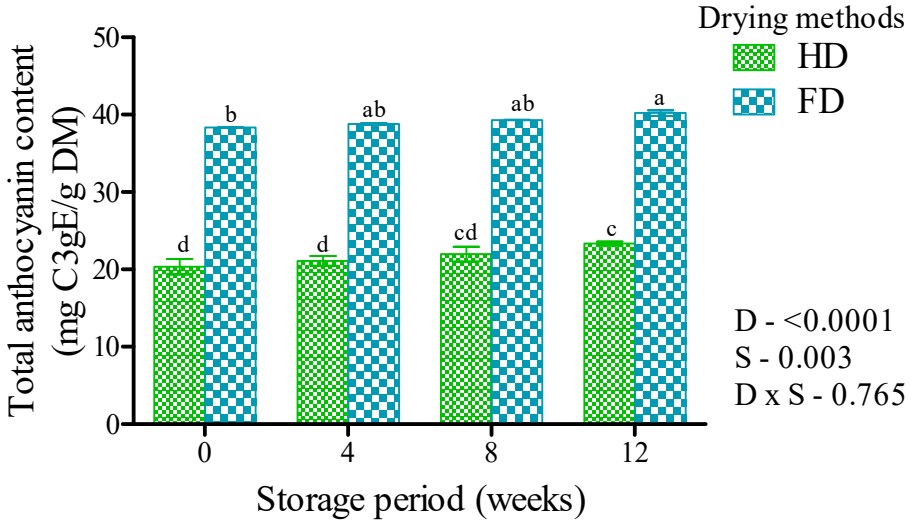

Figure 4. Changes in the total anthocyanin content of pomegranate dried arils during 12 weeks of cold storage at 7 ± 0.3 °C, 92 ± 3% RH (w.b). HD, hot-air drying; FD, freeze-drying. D, drying methods; S, storage period (week). Different letters are significantly different ($p < 0.05$).

3.5. Antioxidant Capacity of Fresh and Dried Arils

The antioxidant capacity of fresh pomegranate arils decreased significantly with storage time from 12.4 to 4.92 mM TE/ 100 mL RSA and 2.36 to 2.07 mM TE/ 100 mL FRAP (Table 3). In relation to their nutritional benefits, phenolic compounds in fruit contribute to the total antioxidant capacity and its subsequent human health benefits [49]. The observed increase in both TPC and TAC was inversely related to the antioxidant capacity (RSA and FRAP) exhibited by pomegranate fruit during storage at 7 ± 0.3 °C, with 92 ± 3% RH. This suggested that antioxidants often react differently depending on the type of antioxidant assay [50]. Siddhuraju et al. [51] reported that a decrease in reducing power could be attributed to the bioactive compounds—total phenolics, flavonoids, ascorbic acids and other hydrophilic antioxidants—associated with the component of the antioxidants present in the fruit.

For dried pomegranate arils, there were significant interactions on the antioxidant capacity (RSA, $p < 0.023$; FRAP, $p < 0.0001$) (Figure 5). The trend showed a general decrease in RSA and FRAP for both hot-air and freeze-dried arils after storage. The FRAP of freeze-dried arils was unchanged at 4 weeks also followed by gradual decline with the storage period (Figure 5) During the storage period, the RSA (26.9 and 29.5 mM TE/g DM) of hot-air dried arils was close to the values reported previously for hot-air dried pomegranate (22.7 to 30.6 mM TE/g) [52] and higher than papaya (9.72 mM TE/g) [53]. Our study showed similar FRAP (2.49–3.27 mM TE/g DM) in freeze-dried arils to those reported for pomegranate cv. Mollar de Elche (3.4 mM TE/g) [54].

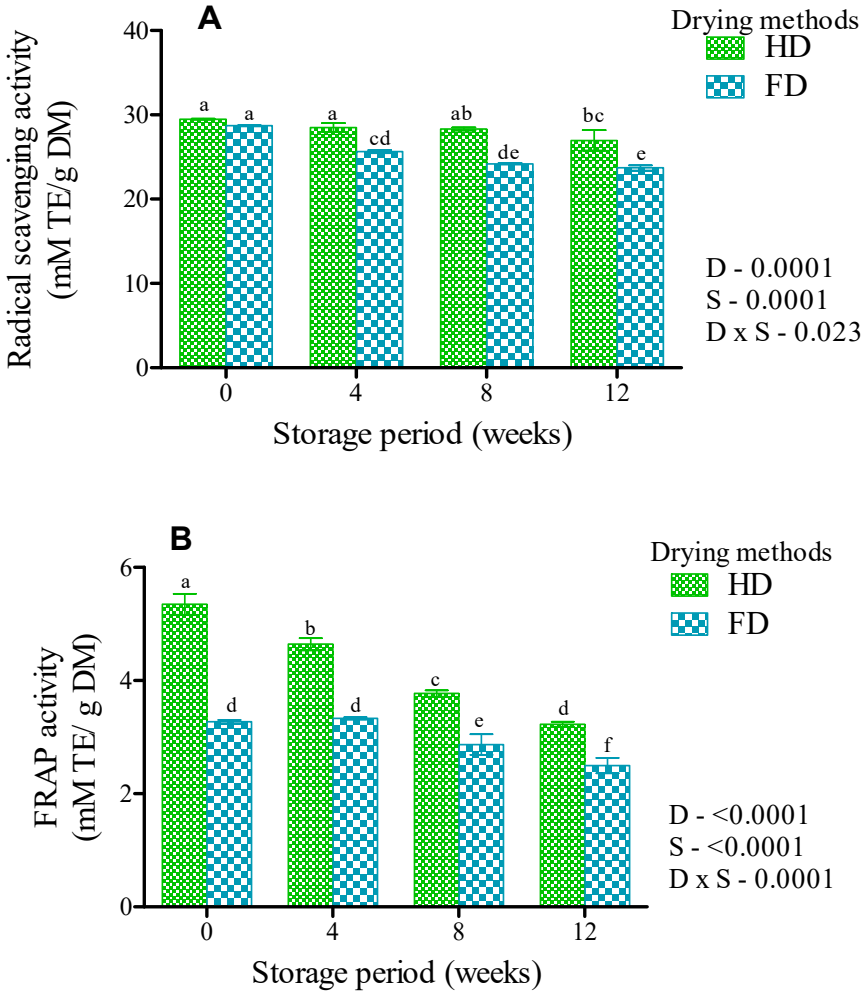

Figure 5. Changes in the antioxidant capacity (**A**) RSA and (**B**) FRAP activity of pomegranate dried arils during 12 weeks of cold storage at 7 ± 0.3 °C, 92 ± 3% RH (w.b). HD, hot-air drying; FD, freeze-drying. D, drying methods; S, storage period (week). Different letters are significantly different ($p < 0.05$).

The decrease in the antioxidant capacity of dried arils could be attributed not only to the same observed decrease in the antioxidant capacity in fresh arils, but also to the thermal degradation of heat-sensitive phenolics—since TPC is reported to be the major contributors to antioxidant capacity. Additionally, Moser et al. [55] reported up to 25% reduction in antioxidant capacity in grape powder after 45 days of storage due to the formation of antioxidant polymers, such as low molecular weight procyanidins. This explanation was also supported by Mrad et al. [56]. However, Michalczyk et al. [57] reported that the antioxidant capacity of dried berries was retained during prolonged storage to a remarkably high degree, which is in contrast with the results from this present study.

Furthermore, freeze-drying amounted to approximately 12.1 and 22.9% lower antioxidant capacity (RSA and FRAP) after the storage period compared to the hot-air dried arils, respectively (Figure 5).

Fracassetti et al. observed a similar decline in antioxidant activity while studying the storage of freeze-dried wild blueberry powder [58]. Mphahlele et al. [2] also reported better retention of antioxidants in the oven drying at higher temperatures 60 °C than in freeze-dried pomegranate peel. The authors attributed this to the concentration of compounds contained in the peel, as these are considered scavengers of free radicals produced during oxidation.

3.6. Stability of Antioxidant Capacity (RSA and FRAP) of Dried Pomegranate Arils

Understanding the stability or degradation mechanisms of food products is essential to maximise the nutritional and sensory quality of products [55]. The stability of antioxidant capacity (RSA and FRAP) of pomegranate arils after hot-air and freeze-drying were evaluated based on changes in their concentrations (Table 4). This table further shows the kinetic parameters (kinetic rate constants and the half-life values) determined for the thermal degradation of the antioxidant capacity. A lower degradation rate indicates lower kinetic rate constants (k) and higher half-life [55].

Table 4. Effect of drying methods on the kinetic parameters of antioxidants (RSA and FRAP) degradation in dried pomegranate arils.

Drying Methods	Antioxidant (mM TE/g)	$k \times 10^{-3}$/(Week^{-1})	$t_{1/2}$/Week	R^2
Hot-air	RSA	0.151	5.654	0.9949
	FRAP	0.129	7.306	0.9949
Freeze-drying	RSA	0.146	5.844	0.9031
	FRAP	0.143	6.597	0.8582

k, kinetic rate constants; $t_{1/2}$; half-life; R^2, coefficients of determination; RSA, radical-scavenging activity; FRAP, ferric ion reducing antioxidant power.

The RSA activity in freeze-dried arils had a lower degradation rate (k = 0.146; $t_{1/2}$ = 5.844) than hot-air dried arils (k = 0.151; $t_{1/2}$ = 5.654); however, the FRAP activity in hot-air dried arils had a lower degradation rate (k = 0.129; $t_{1/2}$ = 7.306) than the freeze-dried arils (k = 0.143; $t_{1/2}$ = 6.597). Considering the calculated degradation k and $t_{1/2}$ as an indicator of the amount of antioxidant loss, with a half-life ($t_{1/2}$/week), the stability of RSA in the hot-air dried arils was approximately 3.3% lower than freeze-dried arils. However, the stability in FRAP activity in freeze-dried arils was 9.7% lower than the hot-air dried arils (Table 4). Several researchers have reported a decrease in the bioactive compounds in fruit after drying [19,59,60]. Zhou et al. [61] reported high degradation in antioxidant capacity (DPPH, FRAP and ABTS) of red pepper.

Similarly, Garau et al. [62] also found that air-drying decreased the antioxidant capacity in orange fruit matrix. This is consistent with the results of this study. Moreover, the values of coefficients of determination (R^2) ranging from 0.85–0.99 were obtained for all linear regressions, indicating that the degradation process of these bioactive compounds for both hot-air and freeze-drying methods followed first-order reaction kinetics.

3.7. Correlations among Quality Attributes for Dried Arils at 12 Weeks of Cold Storage

Significant relationships that exist among attributes measured for dried arils are presented in Table 5.

Pearson's correlation tests indicated a strong positive relationship between TPC and TAC (r = 0.998) (Table 5). Additionally, there were strong negative correlations between TPC and RSA (r = −0.894) as well as TPC and FRAP values (r = −0.998); TAC and RSA (r = −0.910) as well as TAC and FRAP (r = −1.000). However, a positive correlation was found between RSA and FRAP (r = 0.919) (Table 5). A similar result was reported by Cano-Lamadrid et al. [54] between antioxidant ABTS and FRAP in osmotically dehydrated pomegranate arils cv. Mollar de Elche. Strong correlations were found between TSS and phytochemical properties (TPC and TAC), but none of the relationships seems to be

applicable in practice. For instance, a strong correlation (r = 0.937) was found between TSS and TAC (Table 5). In practice, no relevant prediction of dried aril flavour could be made using total anthocyanin content since soluble solids measurement technique applies only to the sweetness (°Brix) of aril tissues. However, a moderately negative correlation was observed between TSS and TA (r = −0.555) (Table 5). This relationship clearly showed that the increase in total soluble solids of dried arils could also contribute to a decrease in TA. Other relationships found a moderately negative correlation between TA and TSS:TA (Table 5).

Table 5. Pearson's correlation coefficients among the investigated parameters of dried pomegranate arils during the 12-week storage period.

Variables	TCD	TSS	TA	TSS:TA	TPC	TAC	FRAP	RSA
TCD	1							
TSS	0.052 *	1						
TA	0.687 **	−0.555 *	1					
TSS:TA	−0.237 ^{ns}	0.941 **	−0.804 **	1				
TPC	0.067 ^{ns}	0.946 **	−0.649 **	0.944 **	1			
TAC	0.122 ^{ns}	0.937 **	−0.612 **	0.922 **	0.998 **	1		
FRAP	−0.132 ^{ns}	−0.944 **	0.599 *	−0.922 **	−0.998 **	−1.000 **	1	
RSA	0.430 ^{ns}	−0.905 **	0.244 ^{ns}	−0.749 **	−0.894 **	−0.910 **	0.919 **	1

95% confidence interval. TPC, total phenolic content; TAC, total anthocyanin content; RSA, radical scavenging activity; FRAP, ferric reducing antioxidant power; TCD, total colour difference; TSS, total soluble solids; TA, titratable acidity. ns; non-significant, * = $p < 0.05$ and ** = $p < 0.001$ (two-tailed).

In consideration of the noted benefits of consuming fruit with high phytochemical properties, it is therefore not surprising that antioxidant capacity (RSA and FRAP) showed a strong positive correlation with TPC. Therefore, the overall quality of dried arils investigated showed that only the interactions among the bioactive components seem promising and practicable.

4. Conclusions

In practice, pomegranate fruit is stored for batch processing. During this time, quality attributes degrade, and hence, the quality of dried pomegranate arils. This study has established the effect of long-term storage of whole fruit on the quality of the final products using hot-air and freeze-drying methods. Prolonged cold storage of raw material considerably affected the total soluble solids and titratable acidity of hot-air and freeze-dried pomegranate arils. The TSS of fresh arils increased while TA decreased with storage period. Freeze-dried aril had a significantly higher total colour difference (TCD) than hot-air dried arils after the 12-week storage period. Hot-air dried arils presented the highest TSS and TA compared to freeze-drying after the storage period. A steady increase in the total phenolic content (TPC) and total anthocyanin content (TAC) of both fresh and dried arils was also observed. Cold storage negatively affected the antioxidant activity (RSA and FRAP) in both fresh and dried arils. At the end of the storage period, freeze-drying presented higher stability of antioxidant capacity (RSA) than hot-air drying.

In contrast, hot-air drying showed higher stability of antioxidant capacity (FRAP) with the highest half lifetime, suggesting that the preservation of antioxidant capacity in dried arils is dependent on the type of assay and choice of drying method. Due to the significantly broad total colour difference (TCD) in fresh fruit after the 12-week storage period, as well as the decline in antioxidant capacity in both the raw material and dried arils in the same period, this study suggests processing fresh pomegranate fruit between harvest and eight-week storage duration. Additionally, based on the importance of colour in marketability and consumer preference, the freeze-drying method is recommended.

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